AD-A053 696 WEAPONS RESEARCH ESTABLISHMENT SALISBURY (AUSTRALIA) F/G 20/2
SOME ASPECTS OF X-RAY TOPOGRAPHY.(U)
AUG 77 R S SEYMOUR
UNCLASSIFIED WRE-TM-1868(A) NL

| OF |
AD
A053 696

END
DATE
FILMED
6-78
DDC

UNCLASSIFIED

(14) WRE-TM-1868(A)

AR-000-922

DEPARTMENT OF DEFENCE

DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION

WEAPONS RESEARCH ESTABLISHMENT

(9) TECHNICAL MEMORANDUM 1868 (A)

(6) SOME ASPECTS OF X-RAY TOPOGRAPHY.

(10) R.J.S. Seymour

(11) Aug 77

(12) 16p.

SUMMARY

The experimental technique of x-ray topography is reviewed for its applicability to the materials characterisation work at W.R.E. Two variations of the technique are critically examined as to the extent of sample covered, resolution attainable and equipment required.

Approved for Public Release

Technical Memoranda are of a tentative nature, representing the views of the author, and do not necessarily carry the authority of this Establishment.

POSTAL ADDRESS: The Director, Weapons Research Establishment, Box 2151, G.P.O., Adelaide, South Australia, 5001.

371 799 JOB

DOCUMENT CONTROL DATA SHEET

Security classification of this page: UNCLASSIFIED

1 DOCUMENT NUMBERS	2 SECURITY CLASSIFICATION
AR Number: AR-000-922	a. Complete Document: Unclassified
Report Number: WRE-TM-1868 (A)	b. Title in Isolation: Unclassified
Other Numbers:	c. Summary in Isolation: Unclassified

3 TITLE

SOME ASPECTS OF X-RAY TOPOGRAPHY

4 PERSONAL AUTHOR(S):	5 DOCUMENT DATE:
R.S. Seymour	August 1977
	6 6.1 TOTAL NUMBER OF PAGES 18
	6.2 NUMBER OF REFERENCES: 9

7 7.1 CORPORATE AUTHOR(S):	8 REFERENCE NUMBERS
Weapons Research Establishment	a. Task: 74/26
	b. Sponsoring Agency:
7.2 DOCUMENT (WING) SERIES AND NUMBER Applied Physics Wing TM-1868	9 COST CODE: 217601

10 IMPRINT (Publishing establishment):	11 COMPUTER PROGRAM(S) (Title(s) and language(s))
Weapons Research Establishment	

12 RELEASE LIMITATIONS (of the document):

Approved for Public Release.

12.0	OVERSEAS	NO		P.R.	1	A		B		C		D		E	

Security classification of this page: UNCLASSIFIED

Security classification of this page: UNCLASSIFIED

13 ANNOUNCEMENT LIMITATIONS (of the information on these pages):

No limitation.

14 DESCRIPTORS:

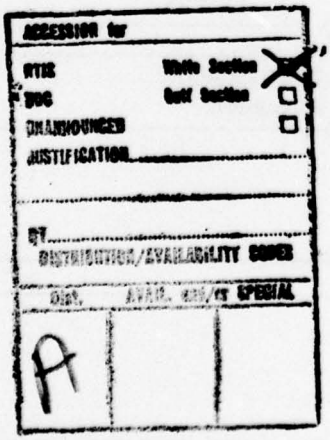
		15 COSATI CODES:
a. EJC Thesaurus Terms	Crystal Structure X-ray inspection Crystal growth Nondestructive tests Crystal defects	2002 1402
b. Non-Thesaurus Terms	X-ray topography Materials characterisation	

16 LIBRARY LOCATION CODES (for libraries listed in the distribution):

SW

17 SUMMARY OR ABSTRACT:
(if this is security classified, the announcement of this report will be similarly classified)

The experimental technique of x-ray topography is reviewed for its applicability to the materials characterisation work at W.R.E. Two variations of the technique are critically examined as to the extent of sample covered, resolution attainable and equipment required.



Security classification of this page: UNCLASSIFIED

TABLE OF CONTENTS

LIST OF FIGURES

1. INTRODUCTION

In a programme involving the growth of single crystal materials, the subsequent preparation of the crystal and ultimately device fabrication, there is an obvious need to evaluate the perfection of the crystals at several stages of the work. Examination of different sections of the crystal boules is desirable to determine where defects originate during crystal growth and this, together with a knowledge of their nature, could lead to the adoption of procedures to prevent such defects. There is also a need to study damage caused in the material by various fabrication processes usually with the aim to minimise such damage. Some particular examples of this type of work envisaged in our laboratory are:

(i) A study of defects introduced during Czochralski growth of a single crystal with the aim of correlating the growth parameters and the appearance of defects.

(ii) A similar study of solution - grown KDP crystals.

(iii) An investigation of the surface damage induced or removed by various mechanical, chemical and ion beam polishing techniques on soft, water soluable crystals such as KDP, and on soft metallic crystals such as tin.

(iv) The examination of the crystalline perfection and composition of epitaxial layers on gallium arsenide and indium antimonide.

A non-destructive method suitable for these studies is x-ray topography (XRT) and some aspects of this technique are described below.

2. PRINCIPLES OF X-RAY TOPOGRAPHY

XRT, as a technique for examining the perfection of, or extent of damage to, large single crystals, was first described by Lang in 1959(ref.1). Extensive use has been made of the technique in the semiconductor industry(ref.2) for such tasks as the determination of dislocation densities in bulk single crystal material(ref.3), the study of the extent of polishing damage(ref.2), and the examination of epitaxial layers(ref.4,5). Of particular interest are studies of growth defects in KDP and ADP(ref.6,7) since these could lead to improvements in growth procedures, bearing in mind that KDP is currently being grown in this laboratory in support of other projects at W.R.E. Examples of the type of information that can be obtained are given in the series of photographs (figures 1-4), taken from the literature.

An x-ray topograph is essentially a picture of a layer of the crystal several microns to a millimeter thick, depending on the x-ray absorption properties of the material. A simple description of the formation of topographs can be given in terms of the familiar Bragg diffraction from a crystal plane, which is described by the relationship

$$2\,d \sin\theta = \lambda$$

Here 2θ is the angle of scattering of an x-ray beam of wavelength λ from a lattice plane of spacing d, inclined at an angle θ to the beam. Consider a broad parallel beam of x-rays incident on a large crystal at the appropriate angle for diffraction by a particular lattice plane, which is not necessarily parallel to the crystal surface. (Figure 5). What is fundamental to the formation of a topograph is the fact that the beam remains parallel after diffraction in accordance with Bragg's Law. Thus the intensity of the screen at say P', Q', R' will

be proportional to the diffracting power of the crystal at P, Q, R. Different parts of the crystal surface will have different diffracting powers if imperfections are present. Examples of imperfections that show up in topographs are local tilting of lattice planes induced by stress, compositional variations which produce local strain and lattice parameter variations, low angle twinning, inclusions of impurities or voids, and all types of dislocations and crystallographic defects. Dislocations are not observable if the atom displacements are perpendicular to the scattering vector (equation(8),(ref.8)), and this fact can be used to determine the direction and nature of the dislocation from a series of topographs from different lattice planes.

The resolution of the x-ray topograph is of considerable interest. A practical limitation is the resolution of the photographic film used to record the diffracted x-rays, and for films which will give adequate exposure in a few hours the resolution is of the order of several microns. Such resolution means that the maximum dislocation density that can be recorded in a crystal sample is of the order of 10^5 to 10^6 equivalent etch pits per square centimeter. It is desirable to ensure that other aspects of the experimental setup do not degrade the resolution even further than the limit set by the recording process. Thus if the divergence of the diffracted x-ray beam is large, then the photographic plate must be very close. A convenient distance is a few centimeters so that the divergence of the diffracted beam should be less than one minute of arc. The divergence of this beam depends on the collimation of the incident beam and the mosaic spread of the sample crystal such that it is less than or equal to the smallest of these. Some highly perfect single crystals have mosaic spreads of a few seconds of arc and if one of these was used as the sample crystal, the collimation of the incident beam would not be important, however for general use it would be necessary to have a well collimated incident beam.

3. TWO METHODS OF OBTAINING TOPOGRAPHS

Of the many experimental techniques that have been described for obtaining topographs, two seem to be most applicable to the type of work envisaged in this laboratory. Both of these enable a large area of sample to be examined with good resolution and both allow either reflection or transmission geometry of the sample to be used.

The first method involves a means of obtaining a parallel beam of x-rays of large cross sectional area and has been described by Kohra et al(ref.9). This method relies on Bragg diffraction from a second crystal with its surface cut at an angle to the diffracting lattice planes as illustrated in figure 6. Here the beam from a x-ray tube is expanded by a factor $b^{-1} = \sin\theta_n/\sin\theta_o$, using the notation of ref.9, where $(\theta_o + \theta_n)/2$ is the Bragg angle θ_B. The angular spread of the diffracted beam is given by $\sqrt{6}$ w where w is the f.w.h.h. of the 'rocking' curve' of the crystal (-its mosaic spread, usually a few seconds of arc). The expanded and well collimated beam is directed onto the crystal of interest, which must first be accurately aligned with the required diffracting planes at the appropriate Bragg angie to the beam. An experimental arrangement adapted from Kohra et al(ref.9), is shown in figure 7.

In the arrangement as actually used by Kohra et al the beam expander was a nearly perfect germanium crystal with (111) diffracting planes selected (Bragg angle 13^o 38'). The crystal was cut at 10^o to the diffracting plane (b = 0.16) and the collimation of the diffracted beam was estimated at 5.8 s. The large distance between the two crystals was necessary to even out, by beam divergence, any irregularities in intensity that would be caused by dislocations in the germanium crystal, and also to give adequate resolution in the vertical plane (see below). The resolution attainable was of the order of several microns, being limited by the resolution of the photographic film, and topographs were obtained with a 90 min exposure time.

A second method of obtaining essentially the same information has been described by Howard and Dobrott (ref.4) and is illustrated in figure 8. Here a collimated x-ray beam of small cross-section is diffracted from the specimen crystal onto a photographic plate. A large surface area is examined by a scanning technique in which the crystal and film are moved together. Slit 2 is adjustable to give the collimation necessary to eliminate $K\alpha_2$ radiation in the final diffracted beam. The collimation required for this can easily be calculated by differentiating Bragg's law to give

$$\Delta\theta = \frac{\Delta\lambda}{\lambda} \tan\theta$$

Hence if $\Delta\lambda$ is to be less than $\lambda(K\alpha_2) - \lambda(K\alpha_1) = \Delta\lambda(K\alpha)$ then $\Delta\theta$ has to be less than the value given by the above formula. For copper radiation, with $\theta = 30^{\circ}$, $\Delta\theta$ has to be less than 5 min of arc, which is achieved by assigning to split 2 a width of 0.5 mm. Such collimation is easily achieved in the double crystal method of Kohra without the use of slits.

The discussion has so far been concerned with the formation of topographs in one plane only (hereafter called the 'horizontal' plane). This is the plane perpendicular to the chosen Bragg planes of both beam expander and specimen crystals in the Kohra method, whilst in the Howard-Dobrott method it is the plane perpendicular to the specimen crystal Bragg plane and the collimating slit. It is in this 'horizontal' plane where either beam expansion or crystal translation is required for coverage of the specimen, but in the 'vertical' plane no such beam expansion or specimen translation is necessary. In this direction adequate coverage of the sample is achieved by merely allowing the x-ray beam to diverge from its point source. X-rays travelling to the crystal surface at the Bragg angle θ to the diffracting plane will travel a distance R and the points of impact with the crystal, projected onto the diffracting plane, will lie on a circle of radius R cos θ. In practice R is chosen large enough such that the portion of this circle on the actual crystal is an approximately linear segment across the entire extent of the crystal in the 'vertical' direction. Any restrictions on the vertical extent of the topograph are only those imposed by the size of the beam expander crystal or by the use of a narrow 'vertical' slit system to produce collimation. In the later case the restriction can be shown to be $2R\sqrt{(\cos\theta\ \Delta\theta)}$ (figure 9). As an example a more than adequate 70 mm extent results from $\Delta\theta = 5'$, R = 1000 mm and $\theta = 30^{\circ}$.

Resolution of the topograph in the 'vertical' direction is not determined by the beam divergence or the mosaic spread of the crystal but rather by the 'vertical' size of x-ray source. As this is of the order of 0.5 mm a source specimen distance of at least 1 m is required in both of the above methods to give a resolution of several microns with specimen-screen distance of centimeters.

4. RECOMMENDATIONS

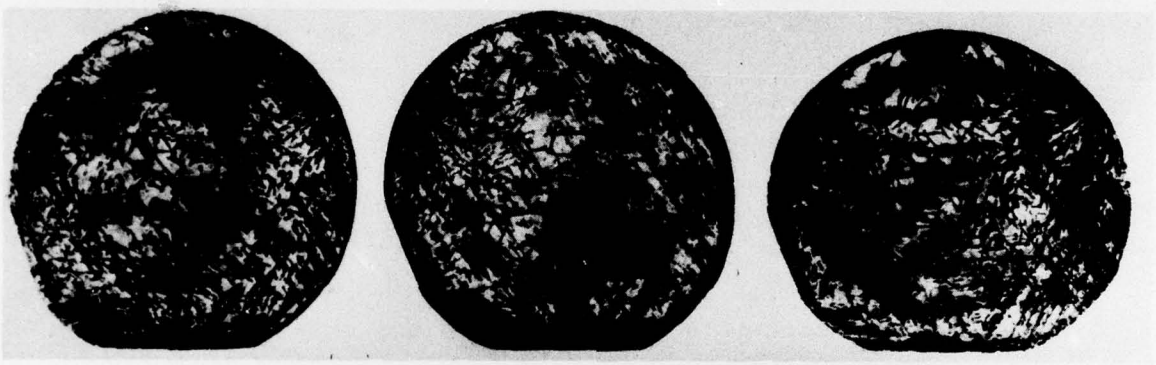
From reports in the literature on the information obtainable by XRT, and in view of its relevance to research into crystal growth and device fabrication, it is imperative that a laboratory engaged in such work should have XRT facilities at its disposal. The major equipment items required are an x-ray source, a beam expanding crystal (with Kohra method), orienting devices for both specimen and beam expander, a photographic film holder, a translation device (with Howard-Dobrott method), and an x-ray detector for initial orientation of both expander and sample crystals. Of these items, this laboratory possesses a suitable x-ray source and a PW1380 goniometer, as well as an x-ray detector which could be used for orienting the beam expander crystal. The remaining components would have to

be constructed and could all be mounted on a separate table, which would be moveable to allow either the Kohra or Howard-Dobrott method to be used. The major piece of equipment that would need to be constructed is an orienting device for the sample crystal, with facility for continuous translation of the sample and film. The beam expander crystal could initially be LiF (200), which is readily grown and prepared in this laboratory. If necessary a highly perfect germanium crystal could later be substituted and the possibility exists of obtaining such a crystal from the AAEC.

REFERENCES

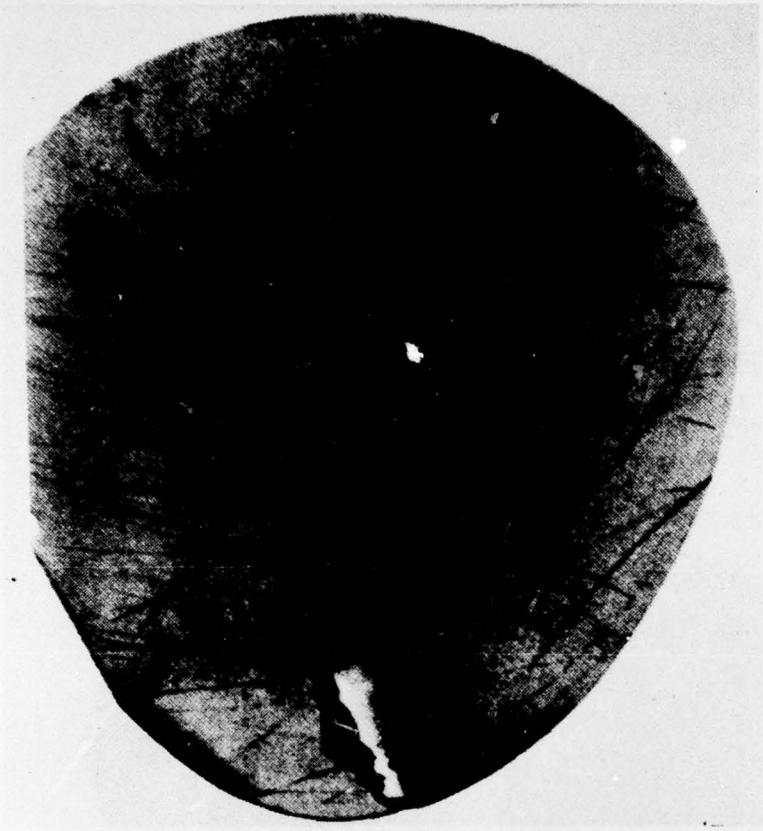
No.	Author	Title
1	Lang, A.R.	Acta Crystallographica Vol 12, p 249, 1959.
2	Kane, P.F. and Larabee, G.B.	"Characterisation of Semiconductor Materials" McGraw-Hill, New York, 1970.
3	Jenkinson, A.E.	Philips Technical Review Vol 23, p 82, 1962.
4	Howard, J.K. and Dobrott, R.D.	"Compositional X-ray Topography" J. Electrochem. Soc., Vol 113, p 567, 1966.
5	Estop, E., Tzrael, A. and Sauvage, M.	"Double-Crystal Spectrometer Measurements of Lattice Parameters and X-ray Topography on Heterojunctions GaAs-$Al_xGa_{1-x}As$" Acta Cryst. Vol A32, p 627, 1976.
6	Belouet, C., Dunia, E. and Petroff, J.F.	"X-ray Topographic Study of Defects in KH_2PO_4 Single Crystals and their Relation with Impurity Segregation" J. Cryst. Growth, Vol 23 p 243, 1974.
7	Deslattes, R.D., Torgesen, J.L., Paretzkin, B., and Horton, A.T.	"Observations of Dislocations in Ammonium Dihydrogen Phospate : Production of Dislocation-Free Crystals" J. Appl. Phys. Vol. 37, p 541, 1966.
8	Seymour, R.S.	"X-ray Technique for Orientation Determination of Large Single Crystals" WRE-TN-1247(AP), 1974.
9	Kohra, K., Hashizume, H., and Yoshimura, J.	"X-ray Diffraction Topography Utilizing Double-crystal Arrangement of (+,+) or Non-parallel (+,-) Setting" Jap.J.Appl. Phys., Vol 9 p 1029, 1970

From ref. 3. Topographs from (a) $(11\bar{1})$ (b) $(1\bar{1}1)$ and (c) $(\bar{1}11)$ planes of a slice of silicon cut (111), showing dislocations. From these photographs the Burger's vector of the dislocations can be deduced.



(a) (b) (c)

Figure 1. Topographs of silicon



From ref.2. Two (440) topographs of a slice of gallium arsenide;

(a) After a polish to produce a mirror finish surface which appeared optically to have no surface damage, and
(b) after a chemical etch to remove another 4.8 μm of material and almost all the surface damage as a result.

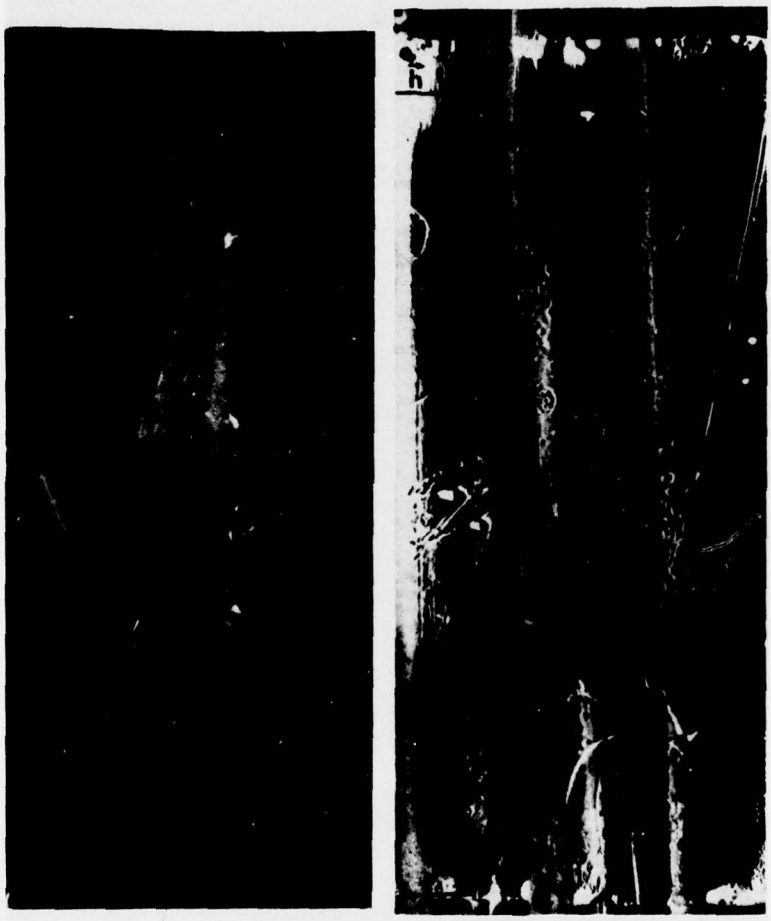
(a)

(b)

Figure 2. Polishing damage in GaAs

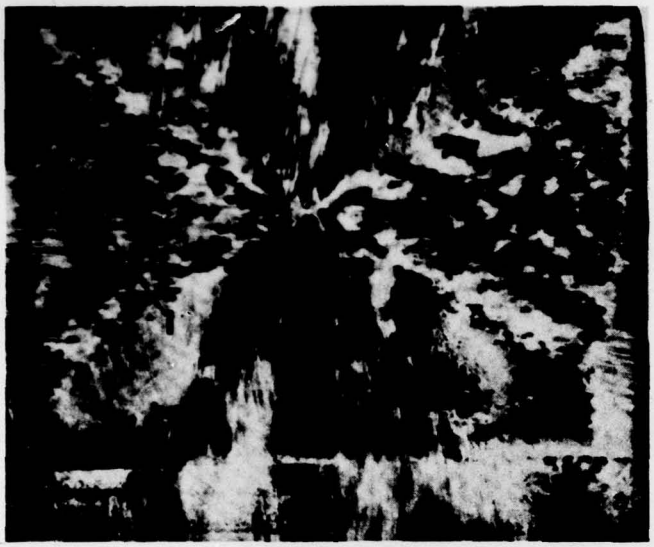
From ref.5.

(a) A topograph $(02\bar{2})$ of a dislocation free crystal of GaAs prior to epitaxy
(b) The same crystal after epitaxy of a 4 μm layer of $Al_{0.7}Ga_{0.3}As$ showing newly created linear defects due to the strain of lattice parameter mismatch.



(a) (b)

Figure 3. Topographs revealing strain in epitaxial layer on GaAs



(a)

(b)

(c)

From ref.6. Topographs of (001) slices of K.D.P. (a) and (b) from near the seed showing dislocations which grow out to the (010) faces as growth proceeds (c) shows contrast from growth layers in their impurity content.

Figure 4. Growth defects in KDP

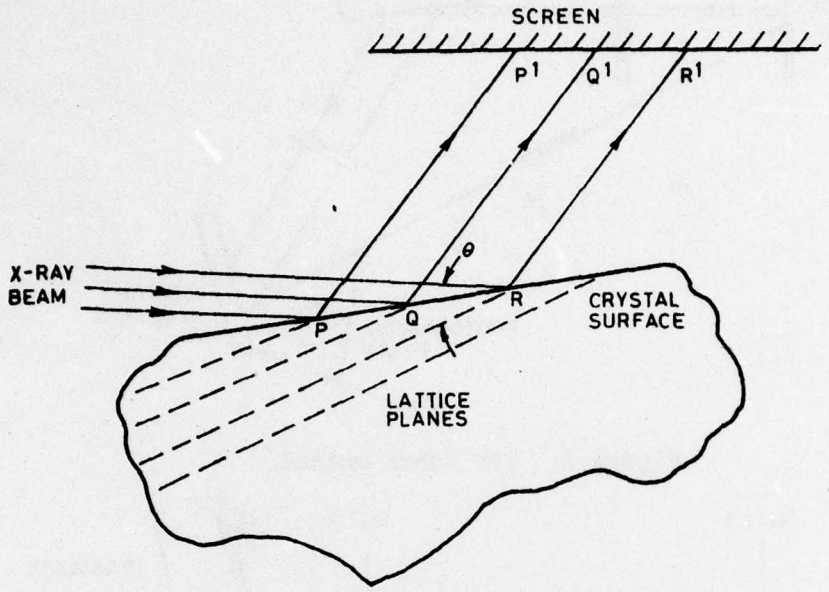


Figure 5. Reflection geometry of Bragg diffraction

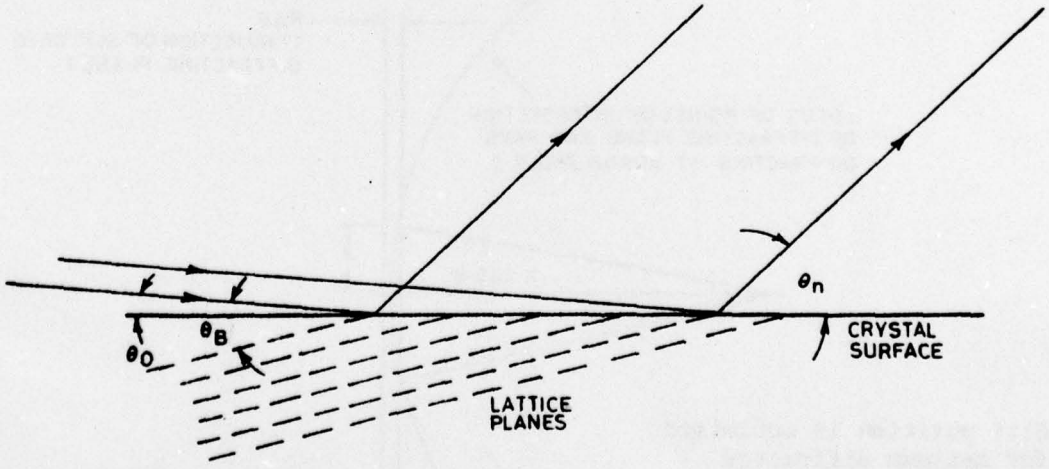


Figure 6. Beam expansion

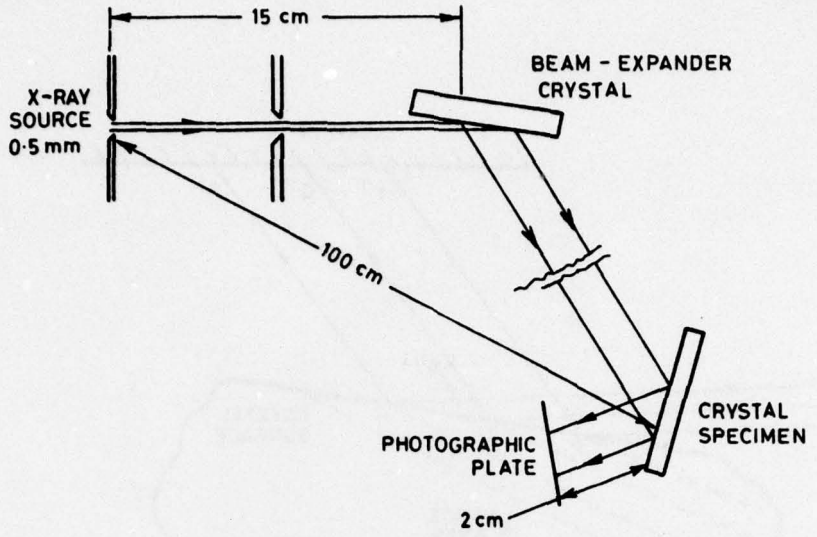


Figure 7. The Kohra method

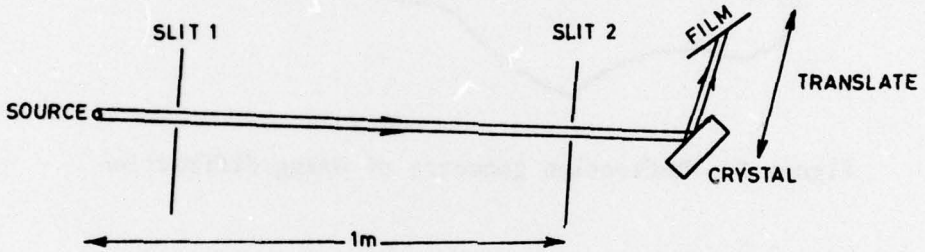
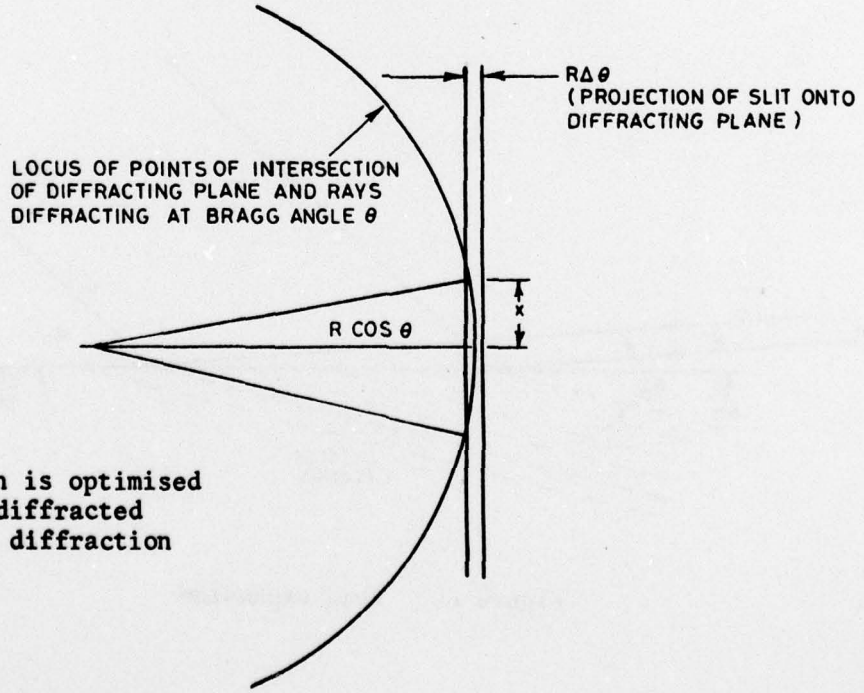


Figure 8. The Howard-Dobrott method



Slit position is optimised for maximum diffracted intensity in diffraction plane.

Figure 9. Derivation of vertical extent of topograph

DISTRIBUTION

EXTERNAL	Copy No.
In United Kingdom	
Defence Scientific and Technical Representative, London	1
In United States	
Counsellor, Defence Science, Washington	2
In Australia	
Chief Defence Scientist	3
Director, Joint Intelligence Organisation (DDSTI)	4
Executive Controller, Australian Defence Scientific Service	5
Superintendent, Defence Science Administration Division	6
Defence Information Services Branch (for microfilming)	7
Defence Information Services Branch for:	
United Kingdom, Ministry of Defence, Defence Research Information Centre (DRIC)	8
United States, Department of Defense, Defense Documentation Center	9 - 20
Canada, Department of National Defence, Defence Science Information Service	21
New Zealand, Department of Defence	22
Australian National Library	23
Defence Library, Campbell Park	24
Library, Aeronautical Research Laboratories	25
Library, Materials Research Laboratories	26
INTERNAL	
Director	27
Chief Superintendent, Applied Physics Wing	28
Superintendent, Optics and Surveillance Division	29
Principal Officer, Laser Group	30
Mr E.H. Hirsch, Laser Group	31
Mr M.F. Penny, Laser Group	32
Mr B.A. See, Laser Group	33
Mr D. Rees, Laser Group	34
Dr R.H. Hartley, Laser Group	35
Dr J. Richards, Laser Group	36
Mr W. Graveney, Laser Group	37
Dr M.A. Folkard, Laser Group	38
Mr W.A.R. Macfarlane, Laser Group	39

	Copy No.
Mr K. Fueloep, Laser Group	40
Mr I. Russo, Laser Group	41
Mr B. Finlayson, Laser Group	42
Author	43 - 46
W.R.E. Library	47 - 48
Spares	49 - 56

	Copy No.
Mr K. Fueloep, Laser Group	40
Mr I. Russo, Laser Group	41
Mr B. Finlayson, Laser Group	42
Author	43 - 46
W.R.E. Library	47 - 48
Spares	49 - 56